

ELECTRON DIFFRACTION IN SODIUM AND POTASSIUM CHLORIDE CRYSTALS*

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Plates XXIII A and B

ABSTRACT. The electron diffraction patterns of NaCl and KCl crystals have been investigated with an electron microscope. The mean values, as calculated from the observed rings, come out to be 5.529 Å for NaCl and 6.22 Å KCl, the standard values being 5.63 Å and 6.28 Å respectively.

INTRODUCTION

In a previous paper, the electron diffraction patterns of different metals, as well as of AgCl and AgBr have been communicated (Majumdar, 1951). In all these experiments, the metals were vaporised in high vacuum on saponin membrane over the objective piece. In the case of halides, silver was vaporised in presence of halogen vapours. It has been found that the spacings as measured from the electron diffraction photographs, agree fairly well with those obtained from X-ray diffraction methods.

In the present investigation diffraction patterns of sodium and potassium chloride crystals have been investigated with electron microscope.

EXPERIMENTAL

The electron microscope was of the Borres-Ruska type (Siemens) and the experiments were carried out at the Virusforschungsinstitut, Heidelberg, Germany. As usual, the projective magnet piece was taken out for diffraction experiments. The objective piece, which was made of platinum-iridium, had an aperture of 0.5 mm. It was placed in water saturated with amyl acetate so that the solution just covered the aperture. A drop of a solution of saponin in amyl acetate was placed on the aqueous solution just over the aperture, when the drop spread itself and covered the aperture. The solution was then drained off and the deposit dried in air. In this way a very thin membrane was obtained on the objective piece. A drop of sodium or potassium chloride solution of different concentrations was next placed on the membrane, and the objective piece replaced within the electron microscope. The vacuum was then turned on. In this way crystals were precipitated on the membrane. A large number of experiments

* This work forms a part of the investigation carried out by the author at Virusforschungsinstitut, Heidelberg, Neckar, Germany in 1950-51.

were done with different concentrations of the salts and also by exposing the crystals to the action of electron beams for different periods of time before taking the photographs. A primary voltage of 160 volts and a secondary voltage of 73 KV in a 9-stage transformer were used in most experiments. For comparison, a few photographs were taken with 70 KV and 56 KV. The photographs of the pictures are given in figures 1-13 of Plates XXIII A and B. The distance between the objective piece and the plate was 57.5 cm.

RESULTS

Table I gives the description of the treatment of the various samples.

TABLE I

Fig. No.	Substance & conc.	Treatment.	Voltage used.
1	NaCl, dil sol.	Taken after 3 min.	73 KV \pm 125 V.
2	"	after 6 min.	"
3	NaCl, 1 mol. sol	after 3 min.	"
4	NaCl, 1 mol. sol	after 6 min.	"
5	NaCl, 1 mol sol.	Immediate exposure.	70 KV \pm 115 V.
6	KCl, dil sol.	Taken after 3 min.	73 KV \pm 215 V.
7	KCl, dil. sol.	after 6 min.	"
8	KCl, satd. sol dil. 1 : 1	Immediately taken	70 KV \pm 215 V
9	"	After longer exposure,	73 KV \pm 215 V.
10	"	Immediately taken.	70 KV \pm 215 V.
11	"	Taken after longer exposure.	56 KV \pm 215 V
12	"	"	"
13	"	Taken immediately.	"

All the diffraction patterns of the same salt with the same high voltage are similar. Certain peculiarities, however, appear with the change of concentration, voltage and the time of exposure to the electron beams before photographing.

The spacings of the crystals were determined by the usual method of trial and error. The distance between the objective piece and plate was 59.5 cm. If $\theta/2$ is the half angle of any particular cone of reflected beams from any particular plane (h, k, l) of the crystal and if x cm is the radius of the particular circle, which represents the section of the cone on the photographic plate, then $\tan \theta/2 = \frac{x/2}{59.5}$, from which $\theta/2$ and $\sin \theta/2$ can be calculated.

For each value of x , the values of $\sin \frac{\theta/2}{\sqrt{1}}$, $\sin \frac{\theta/2}{\sqrt{2}}$, $\sin \frac{\theta/2}{\sqrt{3}}$, $\sin \frac{\theta/2}{\sqrt{4}}$, etc. are

calculated and tabulated. The value of constant K in the equation

$$a_0 = \frac{\lambda}{2 \cdot \frac{\sin \theta/2}{\sqrt{h^2 + k^2 + l^2}}} = \frac{\lambda}{2K}$$

is next determined for a particular value of x , and the mean value of K is taken. λ is then determined from the equation.

$$\lambda = \frac{h}{\sqrt{2m_0 \cdot e \cdot U \left(1 + \frac{e \cdot U}{2m_0 c^2} \right)}}$$

where h = Planck const. = 6.626×10^{-27} erg/sec. ;

m_0 = rest mass of the electron = 9.108×10^{-28} gm.,

e = charge of the electron = 4.803×10^{-10} C. G. S.,

c = velocity of light = 3.00×10^{10} cm/sec.,

U = applied E. M. F. (C. G. S.).

The values of λ for 73 KV and 56 KV are found to be .04386 Å and .0504 Å respectively.

Tables II-IV give the analysis of two typical photographs of NaCl and KCl with 73 KV and one photograph of KCl with 56 KV.

TABLE II

NaCl

Voltage = 73KV \pm 215 volts

$\lambda = .04386$ Å

Nature	S	S	W	W
x (cm)	1.845	2.615	3.200	4.060
$\theta/2$	1°55'	1°18'	1°36'	2°0'
$\sin \theta/2$.0160	.0227	.0279	.0349
K	.00400	.003950	.00398	.00398

Mean value of $K = .00397$. Hence $a_0 = \frac{.04386}{2 \times .00397}$ Å = 5.529 Å.

Standard value of NaCl is 5.63 Å.

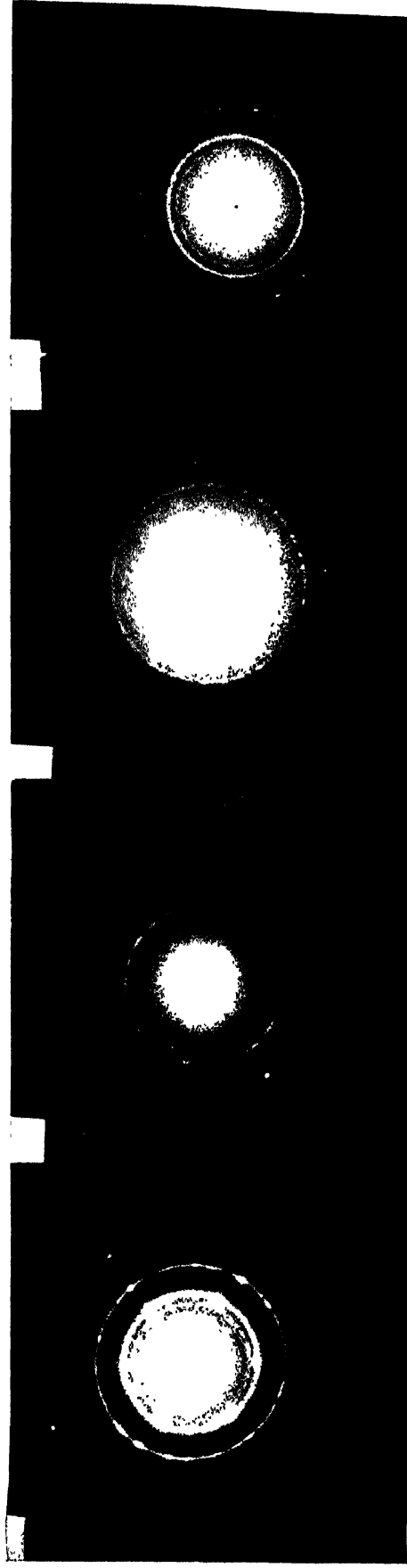


Fig. 1

Fig. 2

Fig. 3

Fig. 4

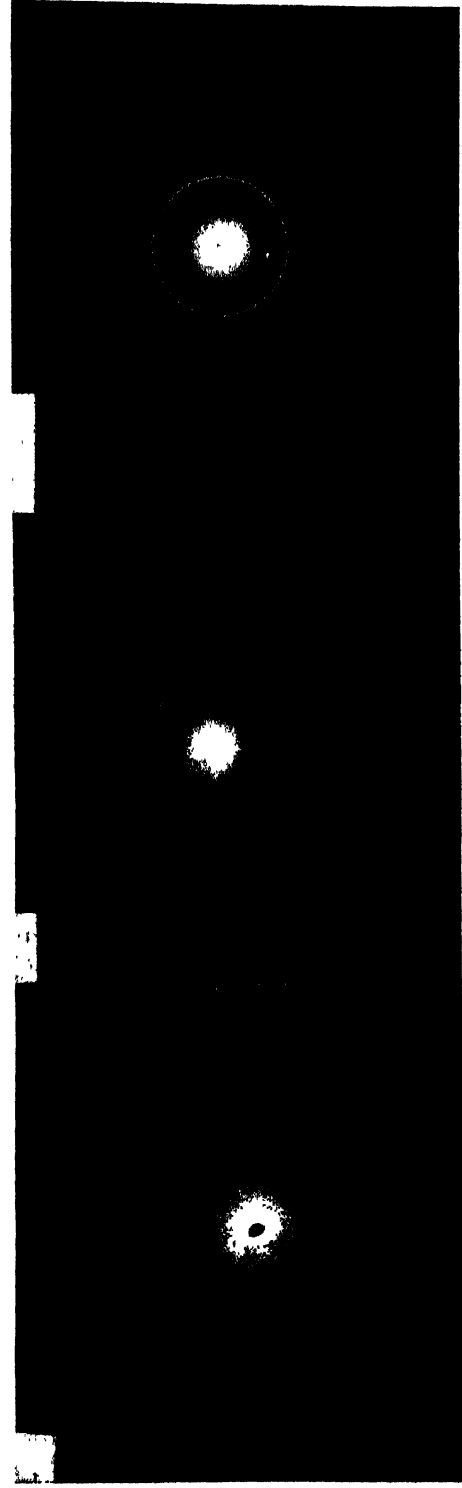


Fig. 5

Fig. 6

Fig. 7

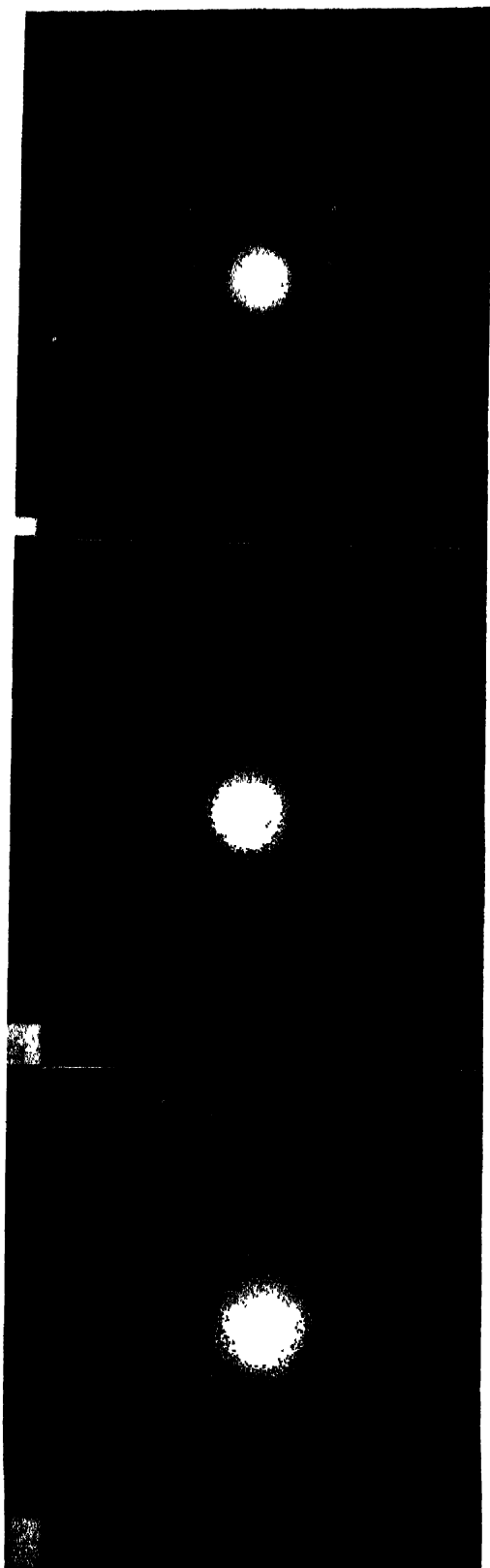


Fig 8

Fig. 9

Fig. 10



Fig 11

Fig 12

Fig 13

TABLE III

KCl.

Voltage = 73 KV \pm 215 $\lambda = .04386 \text{ \AA}$

Nature	S	S	W	VW	W	VW
$x(\text{cm})$	1.602	2.360	2.885	3.450	3.750	4.00
$\theta/2$	$1^\circ 49'$	$1^\circ 11'$	$1^\circ 26'$	$1^\circ 43'$	$1^\circ 52'$	$1^\circ 59'$
$\sin \theta/2$.0144	.0206	.0250	.0300	.0326	.0348
K	.00360	.00363	.00368	.00361	.00360	.00360

Mean value of $K = .00360$ Hence $a_0 = \frac{.04386}{2 \times .00360} \text{ \AA} = 6.09 \text{ \AA}$.

Standard value for KCl = 6.28 \AA

TABLE IV

KCl.

Voltage 56 KV \pm 215 Volts. $\lambda = .0504 \text{ \AA}$

Nature	S	S	W	V. W.	V. W.
$x(\text{cm})$	1.60	2.75	3.25	4.25	4.75
$\theta/2$	$0^\circ 48'$	$1^\circ 22'$	$1^\circ 37'$	$2^\circ 7'$	$2^\circ 22'$
$\sin \theta/2$.0139	.0240	.0282	.0368	.0413
K	.00401	.00400	.00402	.00409	.00413

Mean value of $K = .00405 \text{ \AA}$.

Hence $a_0 = \frac{.0504}{2 \times .00405} \text{ \AA} = 6.22 \text{ \AA}$.

Standard value for KCl = 6.28 \AA .

The values of the spacings of NaCl and KCl thus found agree fairly well with those obtained from X-ray diffraction experiments. An interesting point, however, is the occurrence of spots in some of the pictures. It will be noticed that spots invariably occur with NaCl solutions, whether dilute or strong and also after short or long exposure, whereas, with KCl there are practically no spots with dilute solutions. These points, which are apparently of Laue pattern, do not always indicate cubical symmetry.

Two reasons may be ascribed to the occurrence of these spots. It is possible that some bigger single crystals, in addition to finer ones, are separated on the membrane while the solution evaporates in the vacuum. While the smaller crystals, haphazardly oriented give typical Debye-Scherrer patterns, the bigger ones, which partially cover the objective aperture, give Laue spots.